



# Environment Conscious Ceramics (Ecoceramics): An Eco-Friendly Route to Advanced Ceramic Materials

M. Singh  
QSS Group, Inc., Cleveland, Ohio

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M. Singh  
QSS Group, Inc., Cleveland, Ohio

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# **Environment Conscious Ceramics (Ecoceramics): An Eco-friendly Route to Advanced Ceramic Materials**

M. Singh  
QSS Group, Inc.  
Cleveland, Ohio 44135  
[msingh@grc.nasa.gov](mailto:msingh@grc.nasa.gov)

## **Abstract**

Environment conscious ceramics (Ecoceramics) are a new class of materials, which can be produced with renewable natural resources (wood) or wood wastes (wood sawdust). This technology provides an eco-friendly route to advanced ceramic materials. Ecoceramics have tailorable properties and behave like ceramic materials manufactured by conventional approaches. Silicon carbide-based ecoceramics have been fabricated by reactive infiltration of carbonaceous preforms by molten silicon or silicon-refractory metal alloys. The fabrication approach, microstructure, and mechanical properties of SiC-based ecoceramics are presented.

## **Introduction**

Since the dawn of human civilization, there has always been a delicate balance between the various activities of mankind that utilize resources while expanding the human frontiers and the need to have minimum influence on the ecosystem. The first two hundred years of the industrial revolution essentially solved the problem of production. However, the massive production of goods also generated tremendous amounts of by-products and wastes. In the new millennium, in order to sustain a healthy life in harmony with nature, it will be extremely important to develop various materials, products, and processes that minimize any harmful influence on the environment.

Ceramics have continued to play a key role in revolutionizing industry. Silicon carbide-based ceramics have been utilized since the beginning of the 20th century as heating elements. However, tremendous growth in research and development activities in this area has occurred in the last fifty years. These materials have high strength, good oxidation and corrosion resistance, high thermal conductivity, and good thermal shock resistance. A number of manufacturing approaches have been used to fabricate these materials including hot pressing/hot isostatic pressing, sintering, reaction bonding/reaction forming, polymer pyrolysis, and chemical vapor deposition. Hot pressing and sintering approaches require significant consumption of energy while CVD and polymer pyrolysis techniques generate liquid and gaseous chemical by-products. The reaction bonding technique typically utilizes silicon carbide and carbon powder combined with polymer binders while resin/pore former derived preforms are used in the reaction forming

techniques. The production of silicon carbide powder is energy consuming. The pyrolysis of resin systems produces chemical by-products, which have to be collected for disposal.

Environment conscious ceramics (Ecoceramics) are a new class of materials, which can be fabricated with renewable resources (wood) and wood waste material (wood sawdust). Wood is a “lignocellulosic” material formed by the photosynthetic reaction within the needles or leaves of trees. The photosynthesis process uses sunlight to take carbon dioxide from air and convert it into oxygen and organic materials. Wood has been known to be one of the best and most intricate engineering materials created by nature and known to mankind [1–2]. In addition, natural woods of various types are available throughout the world. On the other hand, wood saw dusts are generated in abundant quantities by sawmills. The environment conscious ceramic materials, fabricated via the pyrolysis and infiltration of natural wood-derived preforms, have tailorable properties with numerous potential applications. The experimental studies conducted to date on the development of materials based on biologically derived structures indicate that these materials behave like ceramic materials manufactured by conventional approaches [3–9]. These structures have been shown to be quite useful in producing porous or dense materials having various microstructures and compositions.

In this study, natural wood has been used to fabricate SiC ceramics through a process of pyrolysis and silicon infiltration as described in previous publications [7 and 10]. The natural internal channels of wood allow the silicon infiltration, and result in a network of SiC after the reaction with carbon. In this work, the microstructure and mechanical properties of SiC fabricated from African Bubinga wood is presented in detail.

## **Ecoceramics Technology**

A schematic of the Ecoceramics fabrication process is given in Fig. 1. The wood pieces were dried in an oven and pyrolyzed in a furnace up to 1000 °C in a flowing argon atmosphere to create carbonaceous preforms. The weight and dimensional changes were recorded after pyrolysis. The pyrolyzed preforms were infiltrated with silicon in a graphite element furnace under vacuum. The infiltration time and temperature depend on the melting point of the infiltrants and dimensions and properties of the preforms. For silicon infiltration, porous preforms were infiltrated at 1450 °C for 30 minutes. A wide variety of wood specimens (softwood and hardwood) and wood saw dusts were used for the fabrication of carbonaceous preforms. These results will be reported elsewhere [11–12]. The ecoceramic technology has been used to fabricate complex shaped parts from the machined wood or carbon specimens shown in Fig. 2.

Although a wide variety of ceramic materials were fabricated using this approach, detailed microstructural characterization and the mechanical properties of SiC fabricated by the infiltration of molten silicon into a pyrolyzed African Bubinga wood preform will be presented. After the infiltration, specimens were machined for microstructural and mechanical property studies. Samples were cross-sectioned and polished for metallographic studies. Microstructural characterization was performed on the as-fabricated and tested samples using optical and scanning electron microscopy. The final product has a cellular structure with elongated areas of SiC and Si.

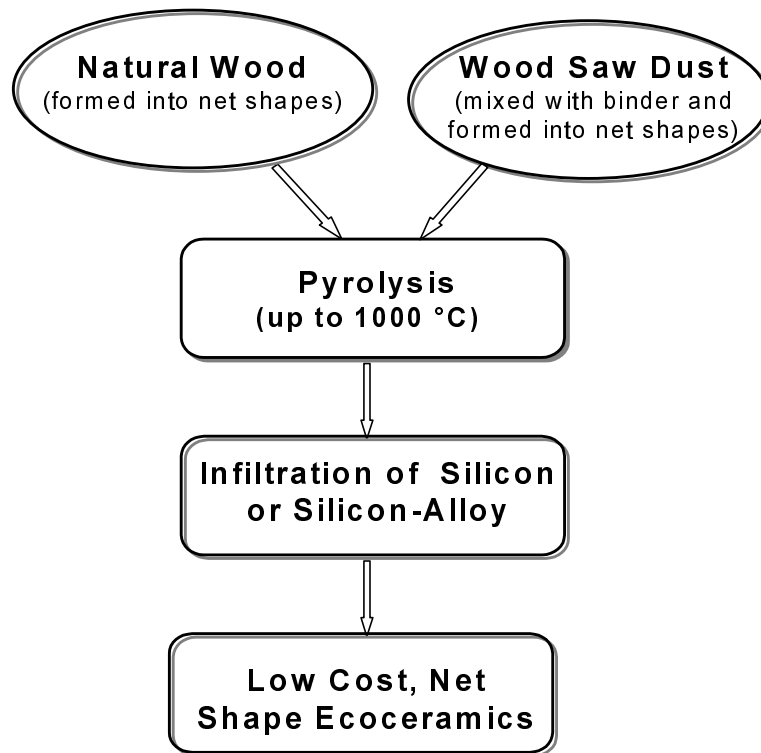


Fig. 1: Schematic of the Ecoceramics fabrication process.



Fig. 2: Photograph showing different shapes fabricated by ecoceramics technology.

## Ecoceramics Properties

A wide variety of wood (softwood and hardwood) specimens were pyrolyzed and infiltrated in this program. Scanning electron micrographs of fracture surfaces of some wood-derived porous preforms are given in Fig. 3. These micrographs show a wide variation in the microstructure and density of the carbonaceous preforms, due to structural differences between various types of wood. The variation of preform microstructure and properties can be utilized to produce final materials with controlled microstructure, composition, and phase morphologies. The pyrolysis shrinkage, composition, and final density of preforms vary greatly depending on the type of wood. The preform density and microstructure control the composition and microstructure of final materials.

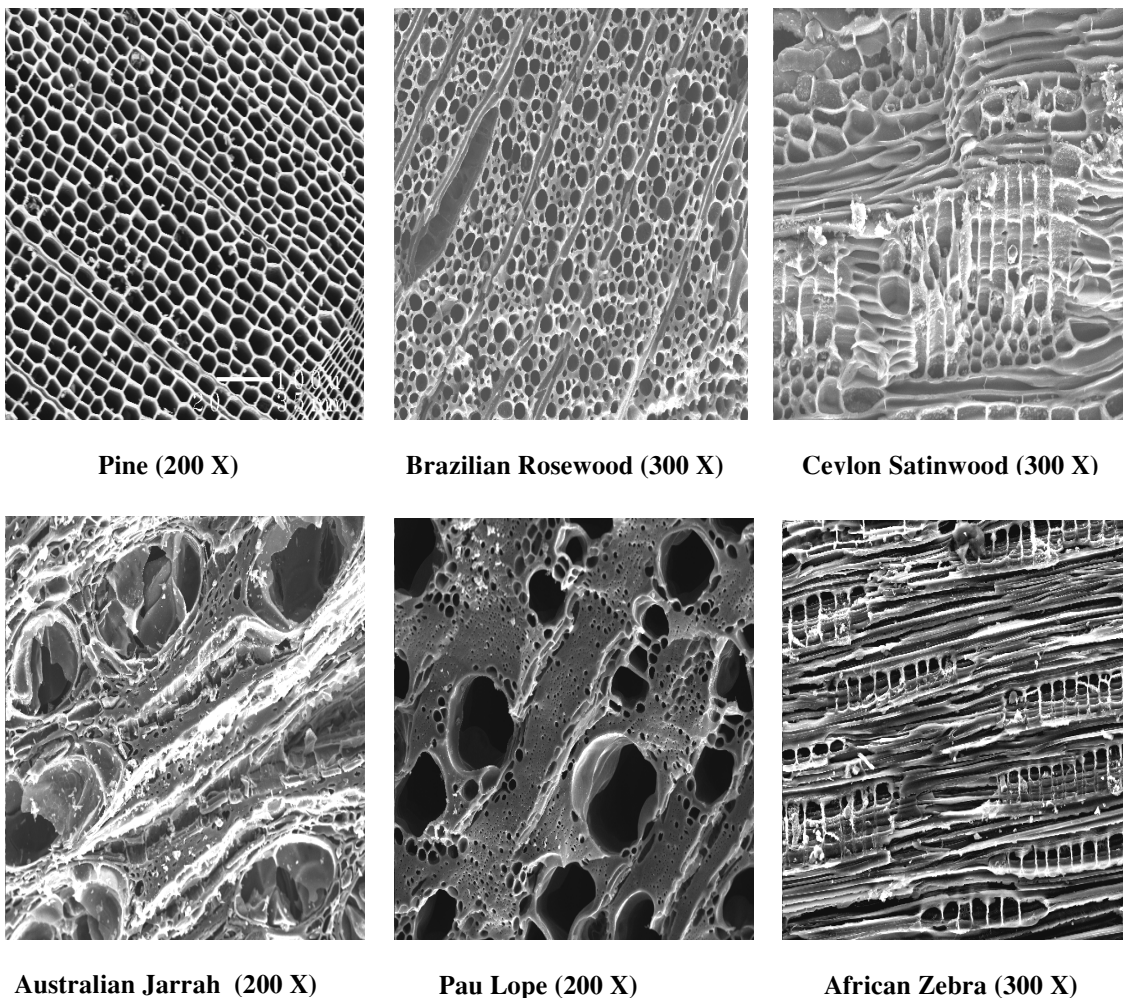


Fig. 3: Microstructure of porous carbon obtained from the pyrolysis of different types of wood.

Microstructure and mechanical properties of a wide variety of wood specimens have been investigated and reported in other publications [7–12]. The African Bubinga wood is from the Leguminosae family of woods and has other common names as Essingang (Cameroon), Ovang,



Kevazingo (Gabon), and Waka (Zaire) [13]. The wood species of this group are found in equatorial Africa from Nigeria through Cameroon to the Congo region. It is found near rivers and lake shores and swampy inundated forests. The tree height is approximately 130 to 150 feet and typically trunk diameters are three to six feet. It is heartwood pink or red brown wood with fine and even texture, with straight or interlocked grains, with a density of 0.65 to 0.78 gm/cm<sup>3</sup>.

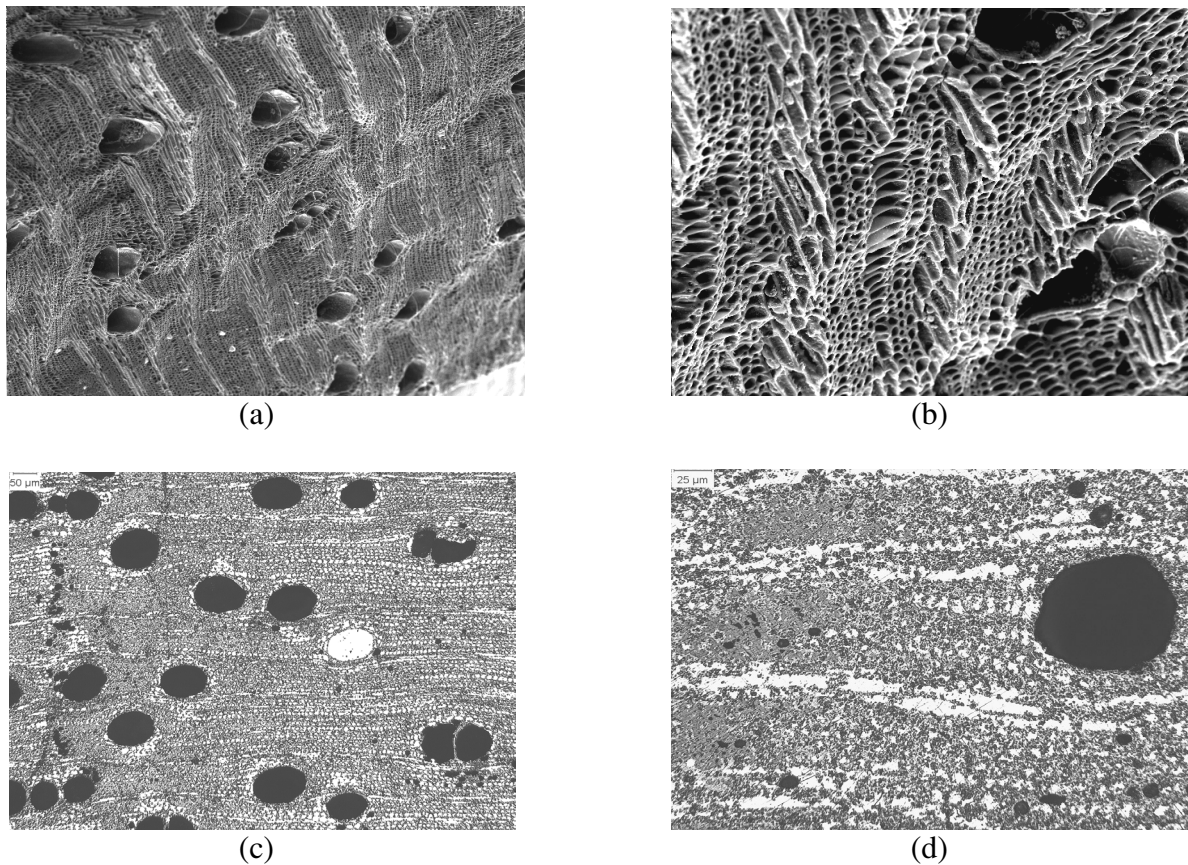


Fig. 4: SEM micrograph of (a) and (b) porous carbon performs (50 and 200 X); (c) and (d) as-fabricated SiC (50 and 200 X) from African Bubinga (white: Si, gray: SiC, black: pores).

Scanning electron micrographs of fracture surfaces of pyrolyzed African Bubinga wood are shown in Figs. 4 (a) and (b). This microstructure shows a heterogeneous pore size distribution (large and small pores). Microstructures of the silicon carbide based materials obtained after silicon infiltration are shown in Figs. 4 (c) and (d). In these micrographs, silicon carbide regions are gray and silicon regions are white. This material also contains porosity (black regions). The density of silicon carbide ceramics characterized in this study was 2.54 gm/cm<sup>3</sup>.

After the melt infiltration, flexure bars were machined from the infiltrated plates. Four-point flexural strength testing was carried out using MIL-STD-1942 (MR) configuration B specimens with 20 mm inner and 40 mm outer spans. Flexure tests were conducted at room temperature,

800, 1200, and 1300 °C in air. Three specimens were tested at each temperature. After testing, fracture surfaces were examined by optical and scanning electron microscopy to identify the failure origins. The room and high temperature flexural strengths of the as-machined materials are shown in Fig. 5. The average flexural strength of as-machined specimens was  $213.3 \pm 26.6$  MPa (RT),  $227.2 \pm 9$  MPa (800 °C),  $217.7 \pm 8.3$  MPa (1200 °C), and  $205.7 \pm 17.9$  MPa (1300 °C). The flexure strength has been compared with a commercially available reaction bonded silicon carbide material (Cerastar RB-SiC). The flexural strength data shows that there is no significant strength loss in these materials up to 1300 °C and it is comparable to commercial RB-SiC materials.

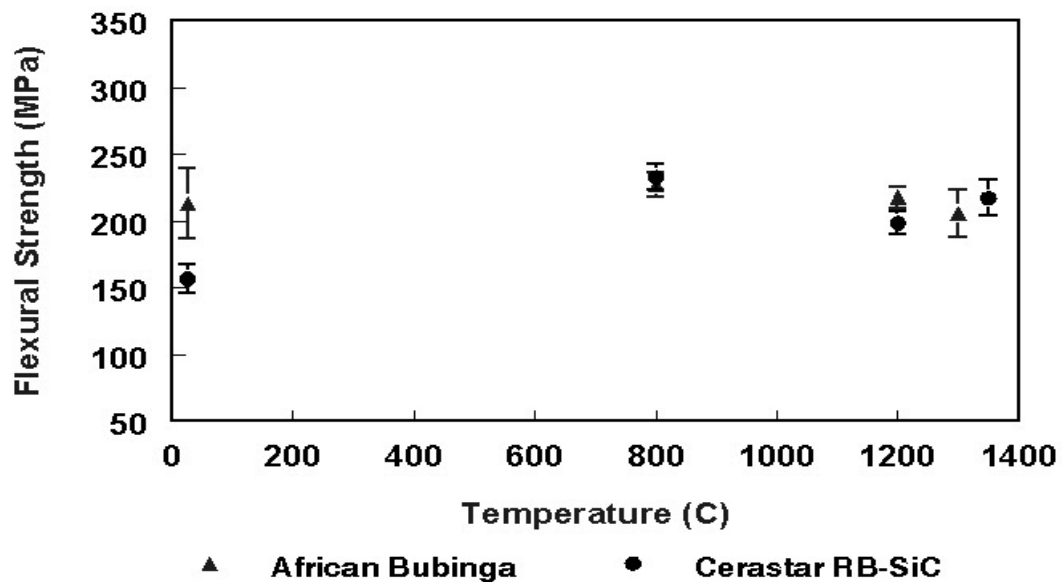


Fig. 5: Flexural strength of ecoceramic made from African Bubinga as a function of temperature.

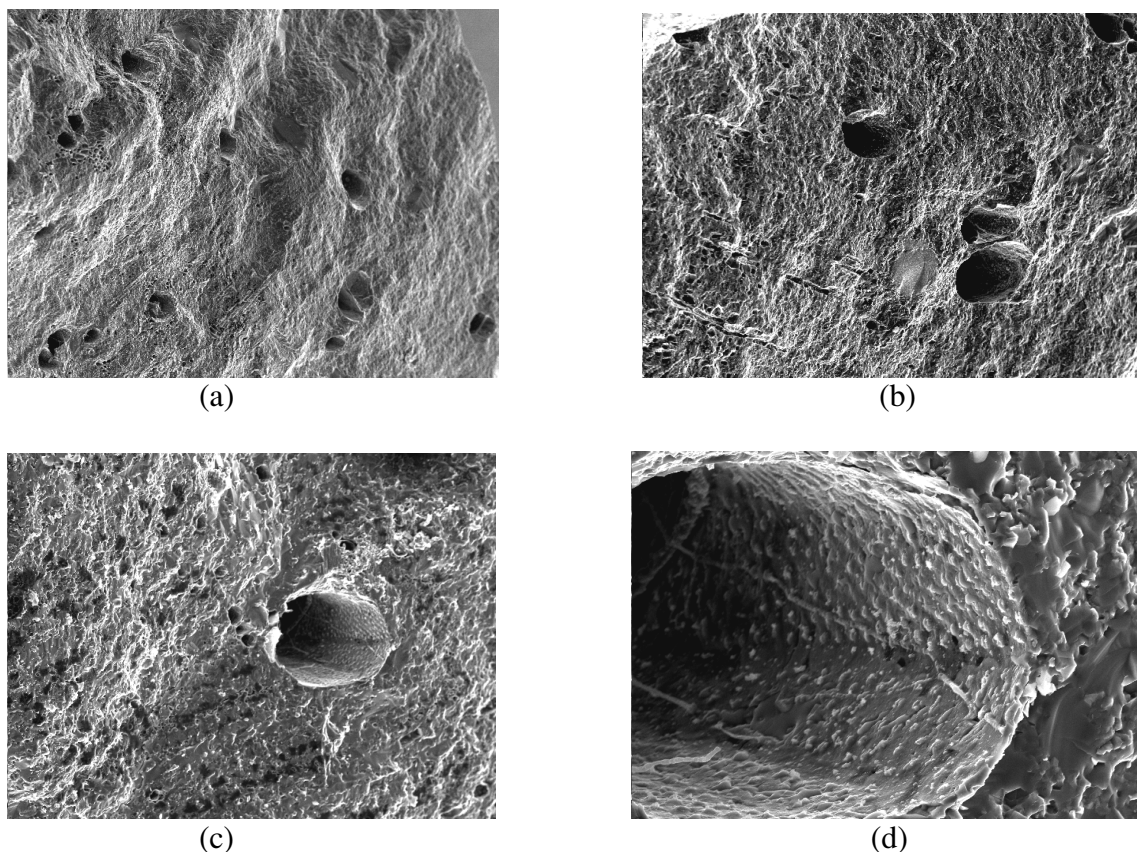


Fig. 6: SEM micrographs showing the fracture surfaces of specimens tested at room temperature (a) 50X and (b) 200X; and at 1300 °C (c) 200X and (d) 800X.

The SEM fractographs of flexure tested specimens are shown in Figs. 6(a)–(d). The fractographs show interesting features. The porosity in these materials was believed to act as the failure origins. Some of these pores were also filled with silicon (Fig. 6(b)). Fractographs of specimens tested at 1300 °C (Fig. 6(d)) show the internal surfaces of pores.

Two of the test methods standardized by ASTM [14] were applied in an attempt to measure the fracture toughness of these materials: the CN (chevron-notch) and the SEPB (Single-Edged-Pre-cracked-Beam) methods. Neither method yielded valid results. In the case of the chevron-notch, stable crack extension, which is required for a valid result, was not obtained. For the SEPB method, the Vickers's indentations that are used to start the through-section pre-crack did not emanate starter-cracks from the indentation corners. This is likely due to the porosity of the material. As a result, a through-section pre-crack for fracture toughness measurement could not be generated without failing the test specimen. Due to the limited number of specimens available, the CN and SEPB techniques could not be investigated in detail in order to determine a solution. Although C 1421 [14] contains a third test method (the surface crack in flexure), the lack of starter-crack formation at indentation sites implied that the technique would not work, and it was not pursued.

In order to measure the fracture toughness, the SEVNB (Single-Edged-V Notched-Beam) method was employed [15]. This technique involves cutting a narrow notch with a small root radius ( $< 0.020$  mm) into a flexure test specimen. The notch tip is sufficiently sharp to produce fracture toughness measurements in general agreement with techniques employing sharp cracks for some ceramics [15]. The fracture toughness of the ecoceramic made from African Bubinga measured  $2.6 \pm 0.5$  MPa m<sup>1/2</sup> (3). Casual observation of the specimen indicated an increase in the fracture toughness with a decrease in the porosity visible on the test specimen surface. The standard deviations are relatively large and may be a result of density variations in the materials. The fracture toughness values for this material are similar to that measured with ASTM C 1421 for other types of silicon carbide [16].

## Conclusions

Environment conscious SiC-based ceramics have been fabricated from renewable natural resources. These ceramic materials have a consistent microstructure that resembles the microstructure of the wood preform. They behave as a silicon carbide-based cellular solid, reaching very high strengths. The low cost, flexibility to fabricate complex shapes, and the availability of unique microstructures in nature makes this fabrication technique very promising for producing materials suitable for structural and lightweight applications.

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